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CENTRAL INTELLIGENCE AGENCY

INFORMATION REPORT

REPORT

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SUBJECT Production of Chemically Pure Calcium in Bitterfeld

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1. The Electrochemical Combine Bitterfeld (the former I.G. plant in Bitterfeld) is completely under Russian control as a Soviet A.G. The greatest secrecy is maintained especially in those parts of the combine which are under Russian supervision and which are looked upon by the Russians as militarily important. The installations for the production of oxalic acid and the calcium installations are especially well guarded.

2. The calcium plant was considerably enlarged in the few months preceding August 1948. The process was changed, so that now metallic calcium is produced there by the fused-mass electrolysis method.

3. In the earlier process, technical calcium was first produced from calcium halogens, especially from calcium chloride, and was then subjected to a refining process in a vacuum. Graphite electrodes were used, which were so arranged that they could be moved in and out of the molten pool. During the electrolysis, these graphite electrodes were slowly removed. Consequently, there was no formation of subhalogens and calcium sponge, which would have interfered with the machining process. In this process, a crust was formed from the electrolyte over the calcium which is unstable in air, and protected the metal from the action of the air. The metal so produced showed a purity of about 98% inside the ingot and (sic) was still contaminated by Si, Fe, Al, Mg and O. These impurities were separated off in the vacuum distillation used in the first stage of the process, and in this manner calcium metal of a purity not more than 99.9% was obtained.

4. This degree of purity is, however, much too low to be used as a reducer for uranium compounds, since even the minutest impurities found in the reducing metal are taken up by the metallic uranium formed in the reduction process. Production of uranium, from which the elements and isotopes important in atomic energy are produced, must be effected so that the uranium does not subsequently require a refining process. The raw material for the production of uranium is uranium pitchblende. According to the well-known process, niobium, tantalum, thorium and the rare earths are first separated off. For this process, large quantities of pure oxalic acid are necessary, since this (acid) makes possible the separation of these accompanying metals, which are separated out as oxalates. As a result, only pure uranium salt (in the form of an oxide) remains, which must be reduced. The greatest difficulties were encountered in reducing these oxides to get pure uranium metal from them.

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5. <u>The new process</u>:

In Bitterfeld a new process has been worked out and is already being employed. With this method calcium metal is produced of a purity which was not possible to attain before. It is based on the fundamental idea that in the production of chemically pure uranium by reduction with calcium in the molten-mass electrolysis, the purest materials must be used. A mixture of halogens no longer serves as the raw material (the material used in starting the process), but <u>pure calcium chloride</u>, which is produced from pure calcium oxide by treating with atomic chlorine. The production of pure calcium chloride is likewise taking place in Bitterfeld. The raw materials calcium oxide and chlorine can be produced in very pure form. The processes employed are well-known. Since chemically pure calcium chloride is obtainable, subsequent impurities in the calcium metal can only come from the other materials which are used in the process.

	Melting Point	Boiling Point
Calcium chloride	approx. 765° C.	approx. 1600° C.
Pure calcium	" 850° C.	" 1440° C.

6. In the Bitterfeld process only one innovation has been introduced: technical calcium is no longer collected at a graphite electrode in order to avoid collecting the impurities in the metallic calcium during the molten-mass electrolysis. The calcium is, on the contrary, produced at a molten copper electrode. During the process a liquid alloy of Cu and Ca is formed. Electrolytic copper is exclusively used, which shows a very high degree of purity. This process takes place at higher temperatures than was the case with the old process, even if carried on in a vacuum.

7. The melting point of pure copper is about 1085° C. If pure calcium is added, the melting point of copper is however progressively reduced as the Ca percentage increases, even down to less than 700° C. Because of this, it is even possible with the new process to work with temperatures of about 900° C. Since this binary Cu-Ca system is eutectic, copper and calcium probably form a compound at the eutectic point. In the new Bitterfeld process, the Cu-Ca alloy is concentrated up to about 60% during the course of the electrolysis. Except for the fact that it might be necessary to interrupt the molten-mass electrolysis for a considerable length of time, the liquid Cu-Ca alloy can be drawn off in a second stage. Then the calcium can be distilled off from the alloy in a partial vacuum, employing protective gases. But only so much calcium is distilled off as to show a calcium content of 30-40% in the remaining alloy. This procedure is chosen because then the remaining alloy still shows so low a melting point that it can be used again by returning it to the electrolytic process in fluid form as an electrode. By means of such a procedure, the procdss can take place indefinitely.

8. The essential advantage of this newly employed process lies in the fact that the impurities normally contained in technical calcium are taken up by the copper in the first part of the process and held there. Consequently, they cannot interfere with the rest of the process. With the vacuum distillation of this new process, calcium of quite extraordinary purity is obtained. The ordinary impurities in calcium metal of Fe, Si, Cl, remained in the refined calcium produced in the previous process in sufficient quantity to interfere with the process. They amount to less than 0.001% in the new process. With this new process, the only difficulties arising are those concerning atmospheric nitrogen, which despite the use of protective gases, cannot be excluded from the process.

9. From a purely stoichiometric calculation, more than two tons of uranium are obtained from one ton of calcium in the reduction of uranium oxide of formula U_3O_8 according to the equation $8\ Ca + U_3O_8 = 3U + 8\ CaO$. The present production in Bitterfeld is between 30 and 40 tons of pure calcium.

10. The finished calcium metal in the form of bars measuring 8-10 cm is se in tin (metal) drums and made ready for shipment to Moscow.